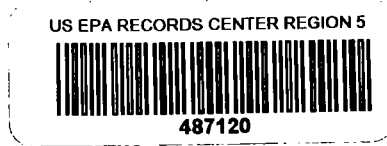


BASF Corporation



BASF

February 28, 2001

Ms. Beth Vens
Environmental Quality Analyst
Environmental Response Division
Department of Environmental Quality
38980 Seven Mile Road
Livonia, Michigan 48152

Subject: Treatability Study Results
 Groundwater from the Riverview Property
 BASF Corporation, Riverview, Michigan

Dear Ms. Vens:

Please find enclosed one copy of the treatability study report concerning groundwater from the Riverview property. Frontier Geosciences, Inc. of Seattle, Washington, performed the study and prepared this report on behalf of BASF Corporation. Portions of this report are referenced in the Feasibility Study, but the report was not appended to the Study.

Please call Mr. Jack Lanigan at 734-324-6219 with questions, or you may call me at 734-324-6209.

Sincerely,

A handwritten signature in cursive script that reads "Thomas F. McGourty".

Thomas F. McGourty
Manager, Safety, Health, and the Environment

Enclosure

cc: Terese Van Donsel, EPA
 Keith Mast, URS (w/out enclosures)
 Jack Lanigan (w/out enclosures)

z:\ecology\offsite\riverview\deq-let37.doc



**FRONTIER
GEOSCIENCES INC.**

ENVIRONMENTAL RESEARCH & SPECIALTY ANALYTICAL LABORATORY

(206) 622-6960 • fax: (206) 622-6870

E-MAIL: info@frontier.wa.com

414 PONTIUS NORTH • SEATTLE, WA 98109

Jack Lanigan
BASF Corporation
1609 Biddle Avenue
Wyandotte, MI 48192

January 2, 2001

Dear Jack,

Following please find my final report covering all activities on the research project entitled "Influence of Speciation on the Efficiency and Reproducibility of Mercury Removal from Contaminated Groundwaters." I have also e-mailed all of the text and graphics to your attention. Please be aware that due to my lack of skill in table formatting and the shortness of time, the e-mailed tables do not exactly match the numbering of the hard copy in all cases. All of the same data is present, however.

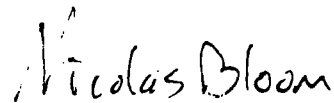
This report includes the contents of the first progress report (sample collection, chemical characterization and the removal of Hg and other metals by pH adjustment with and without the presence of soil particles), as well as all subsequent experiments in the original proposal (APDC extraction, Keylex-100, air stripping, $\text{Fe}(\text{OH})_3$ + carbon, and the leachability of produced sludges). Additionally, an initial optimized two-stage treatment experiment was carried out using one gallon of composited site waste water. Together, these comprise tasks 001-010 on purchase order No. 30262482.

After considering this report, it may be worth while for me to make a presentation of the results to yourself and the appropriate BASF decision makers to answer questions and to design an "optimization mode" micro-pilot-scale experiment to try and treat, for example, 100 litres of composite waste water. This would allow more accurate assessment of reagent costs, sludge generation, and final effluent quality for the technology that is chosen as most promising. I expect that with optimization, effluent concentrations of approximately 10 ng/L Hg will be achievable using an approach as outlined in Table 12. It is hard to

judge the cost of such a system, but owing to the very difficult matrix, it would likely be in the range of \$2 to \$25 per cubic meter treated, with a generation of about 5% of that volume in sludge.

At our last visit, you had also suggested that there was another (easier) waste stream that you would like to have characterized and tested according to these protocols. Please feel free to call or e-mail to discuss these results and make suggestions as you receive the information.

Best Wishes,

A handwritten signature in black ink that reads "Nicolas Bloom". The signature is written in a cursive, slightly slanted style.

Nicolas S Bloom
Sr. Research Scientist

Influence of Speciation on the Efficiency and Reproducibility of Mercury Removal from Contaminated Groundwaters (Final Report)

Nicolas Bloom
Frontier Geosciences Inc.
414 Pontius North, Suite B
Seattle, WA 98109

January 1, 2001

Summary

A series of experiments was undertaken to chemically characterize the groundwater in a contaminated landfill site, and to determine the most promising approach(es) to the treatment of this water for Hg removal, as would be required in a pump-and-treat system. The water was found to be high in Hg and As, as well as sulfide, pH and dissolved organic carbon. Although a small fraction of the total, the samples were none-the-less quite high (near 1 $\mu\text{g/L}$) in methyl mercury, with the remainder of the Hg largely present as dissolved organo-complexed species (most likely humic acid complexes). Arsenic was found to exist as a wide variety of unidentified compounds, very little of which were simple As(III) or As(V).

The raw water is not very amenable to any treatment option, but upon acidification, much of the dissolved organic matter precipitates out, which co-precipitates much of the Hg as well. Typically about 90-99% of the total Hg present was removed by acidification alone, with up to an other order of magnitude removed by secondary treatment options. The best of these, when applied to acidified and settled water was the sulfhydryl resin, Keylex-100, which was able to reduce Hg to 82 ng/L in a static test. It is likely that with further optimization, this could be improved to < 10 ng/L in a column-based (flow-through) extraction scheme. We also demonstrated the potential for *in situ* treatment, based upon oxidation and acidification of the groundwater in place, where the bulk of the Hg and other metals would then simply re-adsorb to the ambient soil particles.

Overall, given the complexity of the starting media, these tests are rather encouraging. Once a particular strategy is selected, it is likely that another factor of 2-10 fold improvement in metals removal will be obtained through fine scale optimization and process control. If both Hg and As were to be removed, however, it is possible that a three-step procedure would be required, because of the very different chemistry of the cationic Hg and the typically anionic As. While the explicit fate of methyl Hg was not investigated in most of the experiments, owing to the high cost of methyl Hg analysis, it is likely that in any chosen treatment scheme it will be destroyed and converted to the more easily removed Hg(II), due to the need for an oxidation step to destroy the sulfides and organic matter prior to treatment.

Introduction

This report covers activities on the research project for the months of October-December, 2000, comprising the successful completion of all of the tasks in the current project. During this time period, Frontier Geosciences sent a sampling team to the Detroit River landfill site in Wyandotte, Michigan to collect analytical and experimental samples from two wells containing water of differing characteristics. During that trip, we also met with BASF personnel to discuss the project, site history, and client needs. Also covered in this reporting period are the results of extensive sample characterization for physical and chemical characteristics, the results from experiments 1-5, which investigated the removal of Hg and other metals from the waters by a variety of means, characterization of the TCLP leachability of project generated sludges, and a simple demonstration experiment (1 gallon) using the optimized parameters uncovered in this study.

Sample Collection

A team consisting of Nicolas Bloom and Lucas Hawkins from Frontier Geosciences Inc. met with Jack Lanigan, a consultant for BASF on October 3, 2000 to visit the site and collect the samples. As a note, Mr. Hawkins' expenses were paid by Frontier Geosciences overhead funds, as a training exercise, and not out of project funds. With the help of Mr. Lanigan, two sample wells, "B" and "D" were selected for sampling, based upon their significant differences in previously measured ancillary chemical parameters, high mercury concentrations, and likely high water flow.

At each well a total of 10 samples were collected, by pumping through Teflon tubing using a peristaltic pump. Each sample set consisted of six 2.5 L samples in borosilicate glass bottles, for experimentation, and four 250 mL samples in borosilicate glass bottles for chemical characterization. One of the four 250 mL samples was field filtered by pumping through a 0.45 μ membrane filtration cartridge. All samples were collected using clean sampling technique (EPA Method 1669), with previously cleaned disposable tubing, filters, and bottles. To collect samples from a well, a 6.5 mm OD length of Teflon tubing was inserted down the well until it stopped, and then pulled back up about 0.5 m prior to pumping. The system was purged by pumping well water through it for one minute (approximately 1,000 mL) prior to commencement of sampling. The samples from each site were numbered sequentially, so that if any temporal changes occurred over the course of purging the wells, they could be noted. The 2.5 L bottles were filled first, followed by the 250 mL bottles. We noted that the water collected was very dark brown in color, contained no visible particulate matter, had an unpleasant organic smell reminiscent of phenolics, and were quite foamy upon agitation.

Also while at the site, a composite surface soil sample was collected. Most of the site was covered with thick grass, and where there were bare spaces, the material was mostly sand. After considerable searching, we found three areas just outside the fence (on the river side) which contained less sandy exposed soil. At each of these sites, we dug up the material with a stainless steel spoon, and sieved it through a 1.4 mm mesh size stainless steel sieve into a clean glass bowl. After approximately one kilogram of fine mesh material was thus collected into the bowl from each site, the soil was thoroughly homogenized with the spoon, making one composite sample of uniform consistency. This material was then used to fill four 500 mL polypropylene jars.

The samples and equipment were taken on the same afternoon to the BASF shipping and receiving area, where they were sent via Federal Express overnight delivery to Frontier Geosciences Inc. They were received by the laboratory by FedEx at approximately the same time as the sampling crew returned from the field, on October 4, 2000. On the day immediately following, sample splits were taken and sent to En-Chem (Madison, WI) and Analytical Resources Inc. (Seattle, WA), the laboratories which were to perform some of the ancillary parameters measurements (see enclosed table). The remaining samples were stored, unopened in a secure locked walk-in refrigerator, which is maintained at a temperature between 0-4°C until they were withdrawn for characterization and experimentation.

Sample Characterization

Three replicates of unfiltered water and the 0.45 μ field filtered aliquot of samples from each well, and the soil sample were analyzed for mercury speciation, and a wide variety of ancillary parameters and ligands known or suspected to affect wastewater treatment efficiency. These analytes and a brief description of the methods employed are reported in Table 1, and so are not repeated here. Overall, where the same parameters were measured, concentrations observed matched the previous analysis from the site quite well, indicating that historic and future analyses from BASF's routine analytical laboratory are sufficient to monitor progress in the remediation of site waters. At some point, if Hg levels are brought low enough ($< 1 \mu\text{g/L}$), it will be necessary to convert to analysis by Method 1631 to accurately trace remedial progress.

Mercury speciation analysis (Table 2) revealed that the site contains little volatile elemental mercury (Hg^0) or dimethyl mercury ($(\text{CH}_3)_2\text{Hg}$), despite seemingly favorable conditions for their formation—high DOC, sulfide, and dissolved organic carbon. These species will not further be considered as part of this investigation. The samples contained high concentrations (in the range of 1 part per billion, or 0.2-3% of the total Hg) of monomethyl mercury (CH_3Hg), which was not unexpected, given our past experience with organic rich alkaline groundwaters. Most of the mercury in the samples, however, appeared to be inorganic Hg(II) , or inorganic mercury that is chelated by organic matter or polysulfides in the samples. Mercury in this form is very difficult to remove by common treatment processes, as the complexes need to be broken, often requiring destruction of the organic matter and sulfides, before treatment of the released Hg(II) can be effected.

We generated some very interesting, and as yet perplexing observations regarding the particle size distribution of suspended colloidal Hg in the well water samples (see Table 6). The field filtered samples indicated that virtually 100% of the mercury from each well sampled was in the dissolved state at the time of sample collection. However, when we performed particle size separations at a range of filter pore sizes on samples in the laboratory, we found that although virtually all of the Hg in well "B" remained dissolved, all the way down to a pore size of 0.1 μ , the mercury in the water from well "D" was 80-100% found on particles smaller than 0.4 μ . Even at the same 0.4 μ pore size used in the field, about 40% of the Hg was on particles, whereas the field filtering

indicated that it was completely dissolved. The difference between the samples is that those filtered in the lab were ones that had been aliquoted for other experiments, so that the water was exposed to an air headspace for approximately a week before the filtering was initiated. At this time we cannot say what occurred in the sample, but oxidation of some of the organics or sulfides may have changed conditions sufficiently to allow the condensation of particulate HgS. No bulk suspended matter was observed to have formed in these samples. Interestingly, we tried to push things along by adding H₂O₂ to further oxidize the samples (1.5% H₂O₂, oxidation for 3 days prior to filtration), and no further increase in particulate Hg formation was seen. The addition of H₂O₂ did clearly oxidize the organic matter, however, as the solution went from nearly black in color to a light tea brown.

Of the other metals tested, arsenic is the most elevated, as had been noted in previous analyses (Table 2). Because we have recently developed an ion chromatography/ ICP-MS technique for arsenic speciation, we decided to analyze the samples by this technique just to see what was there. Although expecting to find mostly arsenite (As(III)) in this reducing environment, we were surprised when the chromatogram emerged with 11 peaks, only the tiniest of which were the common inorganic forms, arsenite and arsenate (Figure 1). The biggest peak belonged to monomethyl arsenic, but many other peaks remain at this time unidentified. On the positive side, we note that organo-arsenic compounds are far less environmentally toxic or regulated than are the inorganic forms. On the negative side, if arsenic removal was ultimately mandated, it might be difficult to accomplish by traditional means, which have been optimized for inorganic arsenic removal.

Antimony is also elevated, in a proportion approximately equal to its rate of co-occurrence with arsenic in the environment. Several other metals, including Al, V, Cr, Ni, and Fe are slightly elevated, but not so high as to present environmental issues. However, the low overall concentration of dissolved transition metals means that when we attempt to use APDC co-precipitation as an Hg removal mechanism, we will need to add a carrier metal in high concentration (20-100 mg/L) to enable the formation of a collectable quantity of the APDC precipitate. The candidate metals for this purpose are Fe, Co, Cu and Zn. Initially we will try Cu, as it forms a very strong APDC bond, but weaker bonds with other complexing agents. If this carrier is found to be successful, but cannot be reduced to low enough levels in the final effluent, we will then try Fe, which, although much less strongly bound by APDC, is also not generally considered toxic in the discharged water.

In addition to confirming previous results, the ancillary parameters analysis (Table 3) revealed several new facts about the ground water. The water was found to be low in Br^- and I^- , which are strong, non-destructible complexers of Hg. Dissolved silica was also low, compared to other alkaline groundwaters we have investigated. Silica builds up, often to percent levels, as the alkaline solution leaches Si from the quartz and clay minerals in the ground. In this case, the low levels of silica suggest that the water in the landfill may not be in intimate contact with very much natural soil, which could be the case if the bulk of the fill material is anthropogenic refuse. Also of importance, the low silica levels may make the pH reduction and settling/filtration scheme a less successful approach than at other alkaline groundwater sites, as this technique relies significantly on coprecipitation of trace metals with the precipitated silica-gel (hydrous silica) that occurs when dissolved silica rich waters are neutralized.

Analysis of the surface soil composite (Table 4) revealed several interesting insights. It is low in Fe and Al, and, for a topsoil, total organic carbon. Given this and the sandy nature of the site soil, I conclude that most of its bulk is made up of silica (quartz), although no silica determination on the soil was made. Other trace metals, except Hg, are within the ranges often seen in surface soils. Mercury was quite elevated at $2.4 \mu\text{g/g}$, compared to $0.2 \mu\text{g/g}$ for typical urban surface soil), as was soil pH at 9.6. When the soil was leached with deionized water 1.7% of the mercury present ($9,600 \mu\text{g/L}$ in the 10:1 leachate—compared to the TCLP acceptance limit of $200 \mu\text{g/L}$) was solubilized. This is certainly due to the high pH of the soil, and by inference, the mode by which the Hg got into the soil. That is, the soil was most likely contaminated by infusion from the groundwater during high-water events, and because of the high pH and apparent low surface area (low relative fraction of clays) of the soil, the Hg is only very weakly adsorbed. This finding is quite unusual as compared to "typical" contaminated sediments and soils. For example, in Hg contaminated surface soils from a site in Peru, total Hg concentrations of up to $50 \mu\text{g/g}$ in the soil yield only $0.001 \mu\text{g/L}$ in the TCLP extracts. Neutralization of the groundwater *in situ*, combined with the use of a higher clay content capping material would dramatically reduce permeability of Hg through this landfill.

Effect of pH Reduction and Soil Addition on Hg Removal

In this experiment six 500 mL aliquots of each of the well waters, and deionized water, as a control, were placed into 1000 mL borosilicate glass bottles.

To three of each water type were added 100 grams of site-derived soil. Because the site well waters were low in transition metals, and we wish to gain information about the general applicability of these treatment methods, all samples were also amended by the addition of 1000 µg/L of Cu to provide additional data. The pH of the samples were then adjusted by the addition of HCl, based upon a previously determined titration, as is indicated in Table 1.5 below. We did not titrate the samples directly with the pH probe in the analytical sample, to avoid potential contamination by the probe itself. Our goal was to obtain values in the range of pH 3-6, but as can be seen in the final tables, this proved elusive for two reasons: the dramatic loss in buffering capacity below pH 6, and the slow kinetics of the acid neutralization due to the addition of soil.

Table 1.5 Titration of 100 mL aliquots of BASF well waters with 12.2 N HCl.

100 mL Well B		100 mL Well D	
HCl (mL)	pH	HCl (mL)	pH
0.00	10.26	0.00	12.77
0.50	9.07	1.00	10.12
0.80	9.07	2.00	7.84
1.00	6.29	2.50	6.54
1.20	5.97	3.00	6.05
1.40	5.41	3.25	4.80
1.50	4.88	3.50	4.71
1.55	3.98	3.60	1.48
1.60	2.35	4.00	1.33

After acidification of the samples with the anticipated amount of acid to obtain pH values in the range of 3-6, the samples were first purged with air for two hours to strip out and/or oxidize compounds such as H₂S and free cyanide, and then agitated overnight on a roller mill at approximately 30 RPM. Tests conducted on the unfiltered sample before and after this treatment verified that no Hg was lost by volatilization. This means that all observed removal of Hg was, indeed due to adsorption on particles and subsequent filtration. The following day, aliquots were filtered through 0.2 µ filters and the filtrates analyzed for Hg, DOC, pH, and select other trace metals (ones either high in the original water, or ones that were low in the water, but might reasonably have been leached into the water from the added soil). A few of these samples were also analyzed for total cyanide.

The results of this experiment are contained in Table 5. We observed that with the addition of soil, over the entire neutralized pH range obtained (pH 4.2 to 7.0), >98% of the Hg, and > 90% of the CH₃Hg, Cu, and Ni were removed to the solids. Final Hg concentrations were in the range of 0.5 µg/L for well "B" water and 2 µg/L for well "D" water. Arsenic was not effectively removed, and some metals such as Cd and Zn actually leached from the soil into the water, albeit at relatively low concentrations. In the samples without soil present, about 98% of the Hg in the well "D" water, but only 30-90% of the Hg in the well "B" water were removed, with greater removal efficiencies occurring at low pH values. Final concentrations for both water types were in the range of 10-15 µg/L. Although Cu was effectively removed by pH reduction only, all of the other metals and CH₃Hg were only moderately removed (0-60% removal), with non-removal for typically oxyanionic species such as Mo, As, and Sb. Upon acidification and filtration, the samples became considerably lighter in color, suggesting the removal of carbon by coagulation, which was borne out by TOC measurements, where concentrations dropped by 30-50%. The limited number of cyanide measurements indicated that despite acidification, little of the cyanide (<50%) was lost, indicating, unsurprisingly, that non-acid-labile stable metal cyanides are present.

This experiment showed that the concentrations of metals in groundwaters could be dramatically reduced by pH reduction, oxidation, and adsorption onto *in situ* soil. Although limits for discharge directly to a water body were not met, it is possible that significantly reducing groundwater mobility in this way could be used as a strategy for leaving the material in place, rather than excavation and/or pump and treat. The technique for effecting this chemistry *in situ* is not fully developed, but companies currently do exist who specialize in pH reduction and oxidation by Fenton's reagent (H₂O₂ + Fe(II) to give OH· radicals) or potassium permanganate (KMnO₄) for the removal of petroleum hydrocarbons. This technology, combined with a more clay-rich capping soil might be applicable in this case.

Direct Treatment of Aqueous Samples

A series of water treatment experiments were simultaneously conducted to maximize analytical resources. These experiments looked at a matrix of three pre-treatment options (no pre-treatment, acidification only, and acidification plus oxidation by H₂O₂ plus UV light) and five treatment options (filtration only, co-precipitation with Cu-APDC, adsorption on Fe(OH)₃ plus powdered activated

carbon, adsorption on the thiol containing resin, Keylex-100, and adsorption on granulated iodinated carbon). Each of these experiments was conducted separately with the waters collected from the "B" well and the "D" well, and two (APDC and $\text{Fe}(\text{OH})_3$) were conducted over a range of reagent additions. Initial pretreatment consisted first of acidification to approximately pH 2 with HCl, and then air stripping for 1 hour to remove any generated free sulfide or cyanide. Following this, the acidified samples were split, and half was treated by the addition of 3% by volume of 50% H_2O_2 , and exposure to a 450 watt Hg-Xe high intensity UV lamp for 1 hour. Over this time, the samples were brought to a full boil by the UV energy absorbed, and 5-10% of the water content was lost. The sample volumes were brought back to the original by the addition of deionized water prior to further treatment steps. At the end of the pretreatment, a total of 6 samples were ready for testing with various treatment technologies: B° and D° (the raw samples), B and D (the pH 2 acidified samples), and B' and D' (the acidified, oxidized samples).

Each of the pre-treated samples was then subjected to the following treatments (since in all cases, the treating agent was removed from the sample by 0.2 μ filtration; any metal removal must be considered in comparison to the filtration-only treatment for that particular pretreatment option). Treatments under a single condition included 0.2 μ filtration, adsorption on Keylex-100 (1 gram per 100 mL) for 18 hours with continuous agitation, and adsorption on granulated iodinated activated carbon (IOC, at 1 gram per 100 mL) for 18 hours with constant agitation. Two treatments were conducted with varying reagent concentrations. The first was coprecipitation by Cu-APDC (copper ammonium pyrrolidine dithiocarbamic acid), which was conducted at 0, 10 and 30 mg/L Cu plus 200 mg/L APDC. These samples were buffered to a pH of approximately 3.5 by the addition of potassium acetate, except for the raw samples, which were treated at ambient pH. The second set were treated with $\text{Fe}(\text{OH})_3$ plus powdered activated carbon (PAC), which was conducted at 0, 10, and 30 ppm Fe, and 0.5 grams per 100 mL of PAC in all cases. In the case of the Fe additions, the pH was adjusted to the range of 9-11 to form the $\text{Fe}(\text{OH})_3$ precipitate. The co-precipitation samples were then allowed to sit with periodic agitation for 18 hours before filtration to remove the solids.

The results of these experiments are shown for all relevant metals in Table 7, as well as in more detail for mercury only in Table 9. Overall, it is clear that most of the mercury removal (99.3%) occurs as a result of acidification plus filtration, with greatest removal efficiencies coming in the case of the oxidized samples at site "B," but with the unoxidized samples at site "D." A very large amount of

suspended matter is generated when these samples are acidified, and the solutions are considerably lightened. It is likely that the generated suspended matter is coagulated humic matter, which scavenges metals under mildly acidic conditions. In all cases, treatment of the raw samples resulted in very poor metals removal (<30%). Overall, best removal efficiencies (>99.9%) were achieved with a combination of acidification plus UV oxidation, together with the addition of Keylex-100.

As an interesting side note, however, the application of activated carbon resulted in effluents which were very clear (removal of organics as well as metals), which could be of value from a public relations standpoint. The use of Cu-APDC in this experiment may have been hampered by the residual oxidizing capacity of the samples after UV photo-oxidation. This may have led to degradation of the APDC complex and subsequent erratic metals removal, which was seen for the Cu carrier, as well as for the Hg. APDC was partially effective at removing As, Ni, and Pb, although for the latter two, initial concentrations were so low as to make removal rates both hard to accurately quantify, and of little site specific importance. None of the methods tried was able to remove more than 30-40% of the arsenic present in these samples.

SnCl₂ Reduction and Air Stripping

Based upon a previous (different site) very successful treatment of Hg in groundwater using SnCl₂ reduction and air stripping of the generated Hg⁰, we attempted this approach on the "B" and "D" waters independently, under several conditions. The air stripping treatment was tested on the raw sample, without SnCl₂ addition, and with the addition of 10 mg/L and 100 mg/L of Sn(II) (approximately 20x and 200x stoichiometric, respectively). Additionally, the treatment was tested on samples acidified to pH 5.5, using 100 mg/L Sn(II). Experiments were conducted using 250 mL of water in tall 500 mL bubbler vessels, using a purge rate of 300 mL of N₂ per minute. All emitted Hg was collected in three intervals (0-30 minutes, 30-60 minutes, and 1-4 hours) by purging the outflow through iodinated carbon traps. These traps were then acid digested and analyzed for Hg content together with the before and after concentrations of Hg in the water samples.

This approach was unsuccessful (0-10% removal) in removing Hg from these water samples, most likely due to the high concentration of Hg-complexing organic matter and polysulfides, which dramatically decreases the kinetics of the

reduction of Hg(II) to Hg⁰. These results are shown in Table 10. It is also possible that the organic matter rapidly complexed the added Sn(II), thereby diminishing the activity of the free Sn(II) in solution. By comparison, in clear, organic free groundwater, we had been able to achieve >99% removal of Hg (albeit at an initial concentration of 0.2 µg/L) in less than 30 minutes of purging with the addition of only 0.5 part per billion (approximately 5x stoichiometric ratio) of Sn(II). The untreated BASF well water samples released more Hg⁰ than did the acidified ones, and more Hg⁰ was released with higher Sn(II) concentrations. It is not surprising that the acidified samples released less Hg⁰, as upon acidification, probably both Hg(II) and Sn(II) became tied up with precipitated organic matter, making them unavailable for reaction.

TCLP Leachability of Treatment-Produced Sludges

All of the solid sludges created were analyzed both for total metals and TCLP leachable metals (Table 8). Total metals were determined on concentrated HNO₃ digests. Because of small sample sizes, TCLP extractions were done in miniature (maintaining the EPA mandated 20:1 liquid to solids ratio), by using 2.0 grams of the sludge, extracted with 40 mL of extractant (pH 4.9 using 0.5M acetic acid + 0.5M potassium acetate). The TCLP samples were extracted for 18 hours with end-over-end tumbling, and then 0.2 µ filtered prior to analysis. In addition to the sludge samples, a sample of 5,000 µg Hg spiked on iodinated carbon was extracted to represent the type of sample that would be obtained if Hg⁰ were purged onto such traps from an SnCl₂ reduction and air stripping application.

Most of the solid phase samples were relatively high in total Hg and As, while the APDC samples were also high in Cu, Sb, Ni, Fe, and V. The Fe(OH)₃ samples were high in Fe, while all other samples were low in all other metals. All samples (except the Hg spiked iodinated carbon) were very low in TCLP leachable Hg and all other trace metals, with the exception of As, which was very easily leached from all of the sludge samples. Because of the high arsenic leachability of these samples, however, it is likely that the sludges would have to be disposed of as hazardous wastes, unless further stabilization was undertaken.

Micro-Scale Treatment Experiment

Based upon the findings in this experiment, a quick micro-scale treatment experiment was undertaken on a one gallon sample consisting of a 1+ 1 mixture

of raw waters from wells "B" and "D." Based on the initial findings, it was clear that both pH reduction and oxidation would be essential for very high level Hg removal from this waste. Thus, the approach chosen was to lower the pH to below 2 with HCl, and then use 0.5% KMnO_4 (a commonly available wastewater treatment chemical) to oxidize the samples prior to secondary treatment. A side advantage of this approach is that the MnO_2 formed in the process may extract additional metals much in the same way that $\text{Fe}(\text{OH})_3$ does. After pH reduction and oxidation, the sample was allowed to settle, and the supernatant treated by one of five approaches: 0.2 μ filtration alone, Co-APDC co-precipitation, 1% Keylex-100, 0.2% powdered activated carbon, and 100 mg/L $\text{Fe}(\text{OH})_3$. In each case, the solids were removed by 0.2 μ filtration, meaning that any enhanced treatment efficiency must be in comparison to that achieved by filtration alone.

The results of this experiment are shown for all relevant metals in Table 11, and the entire experimental summary, including estimated sludge production is presented for Hg in Table 12. The use of pH reduction plus oxidation alone resulted in a 98.7% Hg removal, while filtration boosted this to 99.5%. The best secondary treatment option was Keylex-100, which reduced the final effluent to 82 ng/L Hg, for an overall reduction of 99.98%. The use of PAC as a secondary treatment step reduced the Hg to 170 ng/L, but produced an almost crystal-clear effluent. Co-APDC did not fare quite as well under these conditions (further optimization may be possible), but it did produce very little additional sludge volume. By far the biggest contributors to sludge were (a) the natural organic matter and Mn from the permanganate addition, and (b) the activated carbon addition (this might be optimized to reduce sludge, or granular activated carbon columns might be substituted). Arsenic was not effectively removed by any of the treatment options, although because we were focusing on mercury, further optimization, perhaps in a tertiary step is likely. Nickel, lead, and chromium were also substantially reduced, although none was at an initial level of concern.

Table 1: Summary of Analytical Methods used on BASF Groundwater Treatment Project

Frontier Geosciences Inc. 414 Pontius North, Suite B Seattle, WA 98109
phone: 206-622-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com

analyte	method description	reference method	typical MDL (ug/L)	comments
total mercury	BrCl oxidation, Sn(II) reduction, purge-and-trap, dual amalgamation, CVAFS detection	EPA 1631	0.0002	
ionic mercury	Sn(II) reduction, purge-and-trap, dual amalgamation, CVAFS detection	EPA 1631 (mod)	0.0002	
elemental mercury	purge-and-trap, dual amalgamation, CVAFS detection	EPA 1631 (mod)	0.0002	
methyl mercury	solvent extraction, aqueous phase ethylation, purge-and-trap, isothermal GC separation, CVAFS detection	EPA 1630	0.00002	
dimethyl mercury	purge-and-trap, isothermal GC separation, CVAFS detection	EPA 1630 (mod)	0.00001	
trace metals	inductively coupled plasma mass spectrometry (ICP/MS)	EPA 1638	0.01-1	
major metals	inductively coupled plasma mass spectrometry (ICP/MS)	EPA 1638 (mod)	1-100	
chloride	ion chromatography	EPA 300.0	20,000	En-Chem (Madison, WI)
bromide	ion chromatography	EPA 300.0	200	En-Chem (Madison, WI)
iodide	inductively coupled plasma mass spectrometry (ICP/MS)	EPA 1638	100	values only approximate
sulfate	ion chromatography	EPA 300.0	20,000	En-Chem (Madison, WI)
sulfide	methylene blue colorimetric method		2	
silicon	inductively coupled plasma atomic emission spectrometry (ICP/AES)	EPA 6010	60	ARI (Seattle, WA)
total phosphorous	automated colorimetric	EPA 365.4	500	En-Chem (Madison, WI)
kjeldahl nitrogen	automated colorimetric	EPA 351.1	500	En-Chem (Madison, WI)
weak acid diss cyanide	acidification, diffusion of HCN through membrane into KOH, amperometric detection	EPA 1677	0.5	
total cyanide	UV irradiation, acidification, diffusion of HCN through membrane into KOH, amperometric detection	EPA 1678	0.5	
alkalinity	titration as CaCO3	EPA 310.2	20,000	En-Chem (Madison, WI)
arsenic speciation	ion chromatography coupled to ICP/MS	none	1	experimental
total organic carbon	high temperature combustion, infra-red spectrometric detection	EPA 415.1	1000	En-Chem (Madison, WI)
total suspended solids	0.2 u membrane filtration, drying at 55oC, gravimetry		1000	
total dissolved solids	drying at 105oC, gravimetry		10,000	
pH	electrochemical probe			
sample agitation	30 RPM roller			
filtration	vacuum filtration through disposable 0.2 u nitrocellulose membrane filter			

Table 2: Trace Metals Characterization of BASF Well Waters (Collected October 2, 2000)

Frontier Geosciences Inc. 414 Pontius North, Suite B, Seattle WA 98109

phone: 206-622-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com

sample	Hg	Hg(o)	Hg(II)	DMHg	Blank and MSA Corrected Metals Concentrations: ug/L (ppb)	Be	Al	V	Cr	Mn	Fe	Co	Ni	Cu
Well-B unfiltered bottle #1	23.1				0.950	0.6	1039	612	312	36	3343	-1	751	26
Well-B unfiltered bottle #2	22.9	0.02		<0.0001	0.935	0.1	1845	533	258	53	4003	-2	674	35
Well-B unfiltered bottle #3	21.4					0.3	397	534	248	12	1810	-5	667	13
mean	22.5				0.943	0.3	1094	560	272	34	3052	-3	697	25
SD	0.9				0.011	0.2	726	45	35	20	1125	2	46	11
RSD (%)	4.1				1.1		66.4	8.0	12.7	60.5	36.9		6.7	44.6
Well-B filt	22.7		2.27		1.012	0.2	93	532	252	7	1506	6	661	11
Well D unfiltered bottle #1	633	0.14		<0.0001	1.683	0.2	592	687	138	9	4483	-5	1287	16
Well D unfiltered bottle #2	794	0.10			1.614	-0.1	214	663	119	1	3766	-7	1272	11
Well D unfiltered bottle #3	814					0.3	369	672	121	2	4017	-7	1307	12
mean	747	0.12			1.649	0.2	392	674	126	4	4089	-7	1289	13
SD	99	0.03			0.049	0.2	190	12	10	4	364	2	17	3
RSD (%)	13.3	23.6			3.0		48.5	1.8	8.3		8.9			22.3
Well D filt	1,008		311		1.752	-0.3	270	662	117	0	3600	-6	1125	11
PBW-1	0.02	0.01	0.03	<0.0001	0.005	-0.3	-44	-71	1	-23	-400	22	-42	-48
PBW-2	0.11	0.02	0.11		0.008	-0.4	-19	-84	-6	-24	-351	25	-68	-50
PBW-3	0.13		0.08		0.005	-0.9	15	-88	-7	-24	-378	18	-77	-50
mean	0.09	0.01	0.07		0.006	-0.5	-16	-81	-4	-24	-376	22	-62	-49
SD	0.06	0.01	0.04		0.002	0.3	29	9	5	1	24	3	18	1
eMDL	0.18	0.02	0.12	0.0001	0.005	0.9	88	27	14	2	73	10	55	2
spiked sample	B unfilt			B unfilt	B-filt	B-filt	B-filt	B-filt	B-filt	B-filt	B-filt	B-filt	B-filt	B-filt
spike level	100			0.0366	25.00	500	500	500	500	500	500	500	500	500
sample	23.1			<0.0001	1.01	0.2	93	532	252	7	1506	6	661	11
sample + MS	129.0			0.0354	22.70	484.2	587	1032	748	516	2049	514	1183	512
net	105.9			0.0354	21.68	484.0	494.0	499.6	495.9	509.6	543.0	508.6	521.2	501.3
% recovery	105.9			96.7	86.7	96.8	98.8	99.9	99.2	101.9	108.6	101.7	104.2	100.3
sample + MSD	120.6				25.11	474.9	569	1012	733	509	2029	496	1152	491
net	97.5				24.10	474.7	475.9	479.4	481.3	502.6	522.8	490.1	490.5	480.6
% recovery	97.5				96.4	94.9	95.2	95.9	96.3	100.5	104.6	98.0	98.1	96.1
mean	124.8				23.90	479.6	578.3	1021.6	740.7	512.6	2039.0	505.1	1167.3	501.5
RPD (%)	6.4				10.1	1.9	3.1	2.0	2.0	1.4	1.0	3.7	2.6	4.1

Table 2: Trace Metals Characterization of BASF Well Waters (Collected October 2, 2000)

Frontier Geosciences Inc. 414 Pontius North, Suite B, Seattle WA 98109

phone: 206-622-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com

Blank and MSA Corrected Metals Concentrations, ug/L (ppb)											
sample	Zn	As	Se	Mo	Ag	Cd	Sb	Ba	Tl	Pb	U
Well-B unfiltered bottle #1	102	851	11	102.0	2.3	1.0	13.3	102	0.02	52.4	6.40
Well-B unfiltered bottle #2	125	836	6	95.0	3.2	1.2	13.2	101	-0.01	76.5	6.02
Well-B unfiltered bottle #3	68	824	3	95.2	0.6	0.6	13.7	61	-0.07	27.4	6.13
mean	98	837	7	97.4	2.0	0.9	13.4	88	-0.02	52.1	6.18
SD	29	13	4	4.0	1.3	0.3	0.3	24	0.05	24.6	0.20
RSD (%)	29.3	1.6		4.1	65.4	35.3	2.2	27.0		47.2	3.2
Well-B filt	63	813	8	95.6	1.5	0.4	13.3	53	0.01	19.4	6.08
Well D unfiltered bottle #1	35	14328	4	160.9	0.7	1.0	390	31	-0.01	20.7	0.78
Well D unfiltered bottle #2	19	13998	9	156.4	0.3	0.6	386	21	-0.03	6.1	0.63
Well D unfiltered bottle #3	19	14038	10	158.1	0.3	0.5	394	21	-0.05	6.5	0.67
mean	24	14121	8	158.5	0.4	0.7	390	25	-0.03	11.1	0.69
SD	9	180	3	2.3	0.2	0.3	4.0	6	0.02	8.3	0.07
RSD (%)	38.2	1.3		1.4			1.0	23.2		74.7	10.7
Well D filt	17	14183	10	159.5	1.3	0.5	397	21	0.01	4.3	0.67
PBW-1	1	-37	-9	0.3	0.1	0.0	0.2	-23	-0.39	-3.8	0.01
PBW-2	-6	-40	-8	0.3	0.2	0.2	0.1	-23	-0.43	-3.9	0.00
PBW-3	-6	-35	-12	0.2	0.2	0.1	0.0	-22	-0.41	-3.7	0.00
mean	-4	-37	-10	0.3	0.2	0.1	0.1	-23	-0.41	-3.8	0.00
SD	4	2	2	0.1	0.1	0.1	0.1	0	0.02	0.1	0.01
eMDL	11	7	7	0.2	0.2	0.3	0.2	1	0.07	0.2	0.02
spiked sample	B-filt	B-filt	B-filt	B-filt	B-filt	B-filt	B-filt	B-filt	B-filt	B-filt	B-filt
spike level	500	500	500	500	500	500	500	500	500	500	500
sample	63	813	8	95.6	1.5	0.4	13.3	53	0.01	19.4	6.08
sample + MS	554	1336	498	613.2	534.3	494.2	503.6	565	501.15	523.4	513.70
net	491.1	523.5	489.9	517.6	532.8	493.7	490.3	511.9	501.1	503.9	507.6
% recovery	98.2	104.7	98.0	103.5	106.6	98.7	98.1	102.4	100.2	100.8	101.5
sample + MSD	549	1300	485	604.2	463.3	488.9	493.8	547	505.10	522.4	509.64
net	486.0	486.6	477.6	508.6	461.8	488.5	480.5	493.7	505.1	503.0	503.6
% recovery	97.2	97.3	95.5	101.7	92.4	97.7	96.1	98.7	101.0	100.6	100.7
mean	551.9	1317.9	491.5	608.7	498.8	491.6	498.7	555.8	503.1	522.9	511.7
RPD (%)	0.9	2.8	2.5	1.5	14.2	1.1	2.0	3.3	0.8	0.2	0.8

Table 3: Ancillary Parameters in BASF Well Waters Collected October 2, 2000

Frontier Geosciences Inc. 414 Pontius North, Suite B Seattle WA 98109
phone: 206-622-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com

sample	Ancillary Aqueous Parameters mg/L (ppm)										Tot P
	pH	TDS	TSS	TOC	SO ₄	S	Cl	Br	Si	I	
Well B unfiltered bottle #1				820	620	35.17	1,300	0.74		0.35	28
Well B unfiltered bottle #2				850	620	38.14	1,300	0.76			29
Well B unfiltered bottle #3			1.6								
mean			1.6	835	620	36.66	1,300	0.75		0.35	29
RSD (%)				2.5	0.0	5.7	0.0	1.9			2.5
Well B 0.2 u filtered	10.26	19,900		1,000	600		1,200	0.73	90	0.36	28
Well D unfiltered bottle #1				760	1,700	7.68	630	0.80		0.34	6.8
Well D unfiltered bottle #2				720	1,700	7.93	590	0.81			6.0
Well D unfiltered bottle #3			1.9								
mean			1.9	740	1,700	7.81	610	0.81		0.34	6.4
RSD (%)				3.8	0.0	2.3	4.6	0.9			8.8
Well D 0.2u filtered	12.77	31,000		650	1,700		650	0.80	148	0.33	7.0
10:1 soil extract				93	170		28	0.35		<0.11	2.0
mean blank						0.001			0.1	0.00	
estimated MDL			0.5	1	2.5	0.004			0.1	0.11	
method	electrode	gravimetric	gravimetric	combustion	IC	colorimetric	IC	IC	ICP/AES	ICP/MS	colorimetric
date analyzed			25-Oct-00	16-Oct-00	17-Oct-00	9-Oct-00	17-Oct-00	17-Oct-00	3-Nov-00	31-Oct-00	11-Oct-00

Table 3: Ancillary Parameters in BASF Well Waters Collected October 2, 2000

Frontier Geosciences Inc. 414 Pontius North, Suite B Seattle WA 98109
phone: 206-622-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com

sample	Ancillary Aqueous Parameters, mg/L (ppm)										
	TKN	WAD CN	TOT CN	ALK	B	Li	Na	Ca	K	Mg	Sr
Well B unfiltered bottle #1	37	11.44	14.06	8,600	4.14	0.046	5,480	22.9	47.6	1.89	0.177
Well B unfiltered bottle #2	40			8,600	4.55	0.054	5,906	9.5	55.6	0.62	0.101
Well B unfiltered bottle #3					3.96	0.038	5,481	13.0	43.6	1.14	0.130
mean	39	11.44	14.06	8,600	4.22	0.046	5,622	15.1	48.9	1.22	0.136
RSD (%)	5.5			0.0	7.2	17.4	4.4	46.3	12.5	52.4	28.2
Well B 0.2 u filtered	38			8,700	4.03	0.047	5,366	3.5	45.1	0.456	0.098
Well D unfiltered bottle #1	24	9.18	13.41	17,000	6.89	0.220	11,047	11.0	107.5	0.139	0.098
Well D unfiltered bottle #2	48	9.00	13.10	18,000	6.29	0.212	10,070	11.7	101.9	0.131	0.088
Well D unfiltered bottle #3					6.77	0.228	11,472	16.1	116.0	0.364	0.114
mean	36	9.09	13.26	17,500	6.65	0.220	10,863	12.9	108.5	0.211	0.100
RSD (%)	47.1	1.4	1.7	4.0	4.8	3.6	6.6	21.4	6.5	62.6	13.1
Well D 0.2u filtered	48			18,000	6.13	0.210	9,635	13.1	99.3	0.106	0.085
10:1 soil extract	1.2			320							
mean blank		0.000	-0.001		0.00	0.003	0.69	1.9	-0.2	0.02	0.00
estimated MDL		0.001	0.001		0.23	0.003	0.37	5.5	4.5	0.01	0.00
method	colorimetric	M-1677	M-1678	titration	ICP/MS	ICP/MS	ICP/MS	ICP/MS	ICP/MS	ICP/MS	ICP/MS
date analyzed	11-Oct-00	12-Oct-00	12-Oct-00	12-Oct-00	14-Oct-00	14-Oct-00	14-Oct-00	14-Oct-00	14-Oct-00	14-Oct-00	14-Oct-00

Table 4: Chemical Composition of Surface Soil from BASF (Detroit River) Landfill Site (October 2, 2000)

Frontier Geosciences Inc. 414 Pontius North, Suite B Seattle, WA 98109

phone: 206-622-6960 fax: 206-62-6870 e-mail: nicolasb@frontier.wa.com

sample	pct solids	pH	TOC	Hg	Total Metals Concentrations, ug/g (ppm) as received basis							
					MMHg	Al	Na	Ca	K	Mg	Cr	Mn
BASF Soil rep 1	90.7	9.57	20,000	2.59	0.00239	9,971	8,469	26,543	14,199	1,594	11	183
BASF Soil rep 2				2.43		11,319	8,581	29,226	12,747	2,128	15	281
BASF Soil rep 3				2.29								
BASF Soil rep 4				2.40								
mean	90.7	9.57	20,000	2.43	0.00239	10,645	8,525	27,885	13,473	1,861	13	232
RSD (%)				5.1		9.0	0.9	6.8	7.6	20.3	21.5	29.9
mean blank				0.000	0.00001	10	137	1052	446	6	-24	-3
estimated MDL				0.001	0.00001	2	62	2478	2300	4	2	1

sample	Total Metals Concentrations, ug/g (ppm) as received basis											
	Fe	Ni	Cu	Zn	As	Ag	Cd	Sb	Ba	Ti	Pb	
BASF Soil rep 1	6,135	9.2	13.1	126.2	22.1	0.60	0.32	<2.3	226	0.34	46.6	
BASF Soil rep 2	6,561	9.7	12.9	128.8	21.7	0.56	0.33	<2.3	237	0.32	49.2	
BASF Soil rep 3												
BASF Soil rep 4												
mean	6,348	9.5	13.0	127.5	21.9	0.58	0.33	<2.3	231.5	0.33	47.9	
RSD (%)	4.7	3.7	1.1	1.4	1.4	4.9	2.2		3.4	4.3	3.8	
mean blank	-95	2.3	-5.0	3.2	-0.3	-0.01	0.03	0.7	-3	-0.07	0.03	
estimated MDL	6	1.5	0.1	1.3	0.6	0.04	0.02	2.3	2	0.01	0.02	

Table 5: Removal of Mercury and other Metals from Well Water by pH Adjustment and Soil Addition followed by 0.2u Filtration

Frontier Geosciences Inc. 414 Pontius North, Suite B Seattle, WA 98109
phone: 206-622-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com

Trace Metals Concentrations, ug/L (ppb)															
Sample	condition	pH	TOC	Hg (baf)	Hg	MMHg	V	Cr	Ni	Cu	Zn	As	Mo	Cd	Sb
well D initial	pre-treatment levels				747	1.649	674	126	1,289	1,013	24	14,121	159	0.7	390
well D test #1	well "D" water only	3.81	430	742.8	7.43	0.871	637	108	225	25	-15	16,460	90	0.3	215
well D test #2	well "D" water only	7.50	500	685.3	15.14	1.005	531	136	263	10	-38	15,972	103	0.1	159
well D test #3	well "D" water only	1.59	460	745.9	16.48	1.488	632	89	223	-9	-42	14,384	50	0.2	72.4
well D test #4	well "D" water + soil 1:5	4.2	490		2.45	0.196	111	55	73	84	70	13,645	148	8.2	88.1
well D test #5	well "D" water + soil 1:5	6.9	480		2.09		102	35	79	72	234	11,646	120	17.5	77.9
well D test #6	well "D" water + soil 1:5	6.9	450		1.78	0.180	94	31	102	82	370	11,334	101	25.8	56.2
well B initial	pre-treatment levels				22.50	0.943	560	272	697	1,025	98	837	97	0.9	13.4
well B test #1	well "B" water only	7.83	1,300	20.75	17.62	0.925	577	264	279	542	2	857	95	0.4	12.9
well B test #2	well "B" water only	5.03	710	21.27	11.33	0.744	525	252	260	51	-16	717	66	0.1	9.0
well B test #3	well "B" water only	1.98	430	21.89	2.53	0.381	421	132	213	-9	-23	452	13	0.0	2.6
well B test #4	well "B" water + soil 1:5	7.0	520		0.453	0.010	101	29	63	26	106	1,247	38	1.0	7.9
well B test #5	well "B" water + soil 1:5	6.5	500		0.462	0.001	90	37	95	11	155	1,007	33	3.0	7.0
well B test #6	well "B" water + soil 1:5	6.6	480		0.309	0.010	88	34	97	30	293	1,016	27	4.7	7.2
DI water initial	pre-treatment levels				0.000	0.000	0.0	0.0	0.0	1,000	0.0	0.0	0.0	0.0	0.0
DI water test #1	DI water only		<1		-0.002	0.000	0	-7	-8	915	14	9	3	-0.1	1.3
DI water test #2	DI water only		<1		-0.005	0.000	0	1	-6	873	33	2	-4	-0.1	0.6
DI water test #3	DI water only		<1		-0.003	0.001	0	4	-3	863	1	2	2	-0.1	0.5
DI water test #4	DI water + soil 1:5	6.3	50		0.066	0.001	12	10	65	47	679	95	7	26.7	6.2
DI water test #5	DI water + soil 1:5	6.3	52		0.044	0.001	14	3	50	39	477	136	10	22.2	6.3
DI water test #6	DI water + soil 1:5	6.4	53		0.067	-0.001	13	15	46	40	274	135	11	16.3	7.9
*Samples were spiked with 1000 ppb Cu prior to treatment, to provide an additional metal for study.															

*Samples were spiked with 1000 ppb Cu prior to treatment, to provide an additional metal for study.

Table 5: Removal of Mercury and other Metals from Well Water by pH Adjustment and Soil Addition followed by 0.2u Filtration

Frontier Geosciences Inc. 414 Pontius North, Suite B Seattle, WA 98109
phone: 206-622-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com

		Trace Metals Concentrations, ug/L (ppb)													
Sample	condition	pH	TOC	Hg (bef)	Hg	MMHg	V	Cr	Ni	Cu	Zn	As	Mo	Cd	Pb
well D test #5	well "D" water + soil 1:5	6.9	480		2.11	0.259	107	41	84	82	259	11,987	122	17.7	79.0
well D test #6	well "D" water + soil 1:5	6.9	480		2.07		98	29	74	62	209	11,304	118	17.2	76.7
mean					2.09		102	35	79	72	234	11,646	120	17.5	77.9
RPD					2.0		4.1	16.7	6.5	14.0	10.6	2.9	1.9	1.4	1.5
spike level					10.00	10.00	1,250	1,250	1,250	1,250	1,250	1,250	1,250	1,250	1,250
well D #5 r2 + 1,250 MS					11.39	9.40	1,482	1,357	1,316	1,174	1,452	13,173	1,505	1,226	1,292
net					9.30	9.14	1,384	1,328	1,242	1,112	1,242	1,869	1,387	1,209	1,215
% recovery					93.0	91.4	110.7	106.2	99.4	88.9	99.4	149.5	111.0	96.7	97.2
well D #5 r2 + 1,250 MSD					10.88	10.01	1,492	1,372	1,303	1,170	1,342	13,298	1,500	1,225	1,292
net					8.79	9.75	1,394	1,342	1,230	1,107	1,133	1,995	1,382	1,208	1,215
% recovery					87.9	97.5	111.5	107.4	98.4	88.6	90.6	159.6	110.6	96.6	97.2
RPD					4.5	6.2	0.7	1.1	1.0	0.4	7.8	0.9	0.4	0.1	0.0
reference material					1641-d	DORM-2	1643-d	1643-d	1643-d	1643-d	1643-d	1643-d	1643-d	1643-d	1643-d
replicate 1					7.565	4.832	36.84	20.50	61.00	22.93	93.32	57.56	117.41	6.65	55.50
replicate 2					7.384		37.46	20.32	62.22	21.97	91.80	57.38	117.30	6.52	55.18
BLK-1					0.0094	0.001	-2.7	17.8	13.5	37.1	108.8	-3.6	9.7	0.2	0.3
BLK-2					0.0092	0.001	-2.9	9.6	10.7	54.7	125.3	8.3	14.9	0.3	0.2
BLK-3					0.0094	0.000	-2.9	58.6	34.8	59.1	120.3	-8.4	17.4	0.1	0.3
mean					0.0093	0.001	-2.8	28.6	19.7	50.3	118.1	-1.3	14.0	0.2	0.3
SD					0.0001	0.001	0.1	26.2	13.2	11.7	8.5	8.6	3.9	0.1	0.1
eMDL					0.0003	0.002	0.4	78.7	39.5	35.0	25.4	25.8	11.8	0.3	0.2

*Samples were spiked with 1000 ppb Cu prior to treatment, to provide an additional metal for study.

Table 6: Investigation of Suspended Hg Particle Size Distribution (BASF Well Waters)

Frontier Geosciences Inc. 414 Pontius North, Suite B Seattle WA 98109

phone: 206-622-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com

treatment	filter (μ)	[Hg] $\mu\text{g/L}$ (ppb)		comment
		"B"	"D"	
initial (t=0)	unfiltered	22.5	747	analyzed from sealed bottles
field filtered (t=0)	0.20	22.7	1,008	several days after collection
particulate, (old bottles) rep 1	0.10	0.184	733.8	"old" bottles were 2.5 L jugs which were opened to take samples for other experiments, and so exposed to a 50% air headspace for about one week before this experiment
particulate, (old bottles) rep 2	0.10	0.765		
particulate, (old bottles) rep 1	0.20	0.153	747.8	to a 50% air headspace for about one week before this experiment
particulate, (old bottles) rep 2	0.20	0.198		
dissolved, (old bottles)	0.20	23.36	141.0	all particulate as well as dissolved results are expressed on a $\mu\text{g/L}$ (ppb in the liquid) basis, since the TSS is too low to quantify accurately
dissolved, aerated (old bottles)	0.20	22.99		
oxidized 4 days with 1.5% H_2O_2 , dissolved	0.20	22.26	128.4	
particulate, (old bottles) rep 1	0.45	0.424	321.6	
particulate, (old bottles) rep 2	0.45	0.385		
particulate, (old bottles) rep 1	1.00	0.351	166.7	
particulate, (old bottles) rep 2	1.00	0.356		
particulate filter blank	0.10	0.003	0.003	
particulate filter blank	0.20	0.001	0.001	
particulate filter blank	0.45	0.014	0.006	
particulate filter blank	1.00	0.002	0.008	
mean		0.005		
SD		0.005		eMDL = 0.013 $\mu\text{g/L}$
particulate, (old bottles) + 0.769 $\mu\text{g/L}$ MS	1.00	1.105		97.4% recovery
particulate, (old bottles) + 0.769 $\mu\text{g/L}$ MSD	1.00	1.102		97.0% recovery (0.3% RPD)

Tables 7 and 8: Metals Removal Summary (BASF wastewater treatment, tasks 2-5)

analyzed by

Frontier Geosciences Inc. 414 Pontius North, Suite B Seattle, WA 98109

phone: 206-622-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com

Waste	pH	Sample	Metals Concentrations: µg/L (ppb)								
			V	Cr	Fe	Ni	Cu	As	Sb	Hg	Pb
B	2.04	acidified, unfiltered	606	322	3,232	355	17	846	13.8	22.77	88.6
B	2.04	acidified, 0.2 µ filtered	450	150	2,406	290	33	528	2.7	1.98	21.3
B	3.75	acidified, 0 ppm Cu, 200 ppm APDC	390	116	1,751	127	-22	733	4.7	2.37	8.7
B	3.54	acidified, 10 ppm Cu, 200 ppm APDC	389	140	1,707	15	-17	725	4.1	1.27	11.7
B	3.24	acidified, 30 ppm Cu, 200 ppm APDC	415	124	871	185	5136	689	3.1	1.05	17.4
B	11.29	acidified, 0 ppm Fe, OH, 0.5% PAC	612	312	2,631	316	21	862	14.1	14.23	40.1
B	11.15	acidified, 10 ppm Fe, KOH, 0.5% PAC	610	300	11,491	319	30	873	14.4	14.76	62.8
B	10.93	acidified, 30 ppm Fe, KOH, 0.5% PAC	605	319	29,223	324	28	868	15.2	15.26	56.0
B	4.30	acidified, 1% iodated carbon	329	142	1,914	208	40	681	3.5	1.39	35.3
B	3.76	acidified, 1% Keylex	248	102	965	195	21	523	1.6	0.81	13.5
B'	2.04	UV photo-oxidized, unfiltered	571	283	2,713	336	33	818	13.0	18.53	66.1
B'	2.04	UV photo-oxidized, 0.2 µ filtered	597	288	1,917	344	30	857	12.6	0.16	57.8
B'	2.91	UV photo-oxidized, 0 ppm Cu, 200 ppm APDC	560	279	2,248	271	29	773	14.0	0.49	44.3
B'	2.92	UV photo-oxidized, 10 ppm Cu, 200 ppm APDC	584	269	1,648	106	-4	798	12.2	0.55	28.2
B'	2.87	UV photo-oxidized, 30 ppm Cu, 200 ppm APDC	551	251	1,410	77	49	768	11.8	0.43	28.3
B'	9.10	UV photo-oxidized, 0 ppm Fe, KOH, 0.5% PAC	559	283	1,028	100	15	823	12.3	0.03	22.8
B'	8.81	UV photo-oxidized, 10 ppm Fe, KOH, 0.5% PAC	563	247	1,143	88	15	819	13.2	0.03	18.4
B'	8.08	UV photo-oxidized, 30 ppm Fe, KOH, 0.5% PAC	576	250	620	72	17	772	12.3	0.03	21.0
B'	4.19	UV photo-oxidized, 1% iodated carbon	571	236	1,169	285	-6	837	10.6	0.16	4.2
B'	2.76	UV photo-oxidized, 1% Keylex	421	248	988	335	22	848	12.1	0.02	65.3
B°	10.26	raw, unfiltered	560	272	3,052	697	25	837	13.4	22.50	52.1
B°	10.26	raw 0.2 µ filtered	532	252	1,506	661	11	813	13.3	22.70	19.4
B°	10.04	raw 0 ppm Cu, 200 ppm APDC	604	262	1,841	197	-9	835	14.1	24.29	18.7
B°	10.03	raw 10 ppm Cu, 200 ppm APDC	569	247	1,637	105	2548	850	14.5	21.11	19.3
B°	10.01	raw 30 ppm Cu, 200 ppm APDC	593	259	1,758	132	4642	706	11.0	5.48	10.7
B°	10.06	raw 0 ppm Fe, KOH, 0.5% PAC	553	259	1,571	263	20	845	14.1	18.68	31.3
B°	10.03	raw 10 ppm Fe, KOH, 0.5% PAC	538	249	4,588	260	13	833	13.2	17.25	20.8
B°	10.01	raw 30 ppm Fe, KOH, 0.5% PAC	522	246	8,595	250	22	820	12.6	15.88	29.2
B°	10.04	raw 1% iodated carbon	585	272	1,936	307	7	842	12.6	20.58	20.2
B°	10.04	raw 1% Keylex	520	293	1,753	372	27	842	14.2	7.63	38.3

Tables 7 and 8: Metals Removal Summary (BASF wastewater treatment, tasks 2-5)

analyzed by

Frontier Geosciences Inc. 414 Pontius North, Suite B Seattle, WA 98109

phone: 206-622-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com

Waste	pH	Sample	Metals Concentrations, $\mu\text{g/l}$ (ppb)								
			V	Cr	Fe	Ni	Cu	As	Sb	Hg	Pb
D	1.93	acidified, unfiltered	688	137	3,849	314	20	14,276	773	787.2	21.1
D	1.93	acidified, 0.2 μ filtered	526	90	2,991	214	17	8,908	80	7.42	26.0
D	3.48	acidified, 0 ppm Cu, 200 ppm APDC	494	63	2,738	12	22	7,948	51	7.59	94.4
D	3.29	acidified, 10 ppm Cu, 200 ppm APDC	503	64	2,792	9	9	8,585	55	2.89	31.5
D	3.91	acidified, 30 ppm Cu, 200 ppm APDC	528	63	483	97	1871	9,494	135	6.27	39.4
D	9.88	acidified, 0 ppm Fe, KOH, 0.5% PAC	628	70	3,573	192	44	13,698	517	15.01	28.9
D	9.86	acidified, 10 ppm Fe, KOH, 0.5% PAC	675	50	3,011	179	-16	14,469	559	18.68	0.0
D	9.69	acidified, 30 ppm Fe, KOH, 0.5% PAC	678	51	3,347	175	12	14,828	548	14.89	0.7
D	4.00	acidified, 1% iodated carbon	344	61	3,194	179	32	9,456	106	2.33	24.9
D	3.49	acidified, 1% Keylex	313	65	1,283	151	15	9,063	58	1.10	17.1
D'	3.27	UV photo-oxidized, unfiltered	645	127	3,467	299	32	13,689	656	531.4	15.4
D'	3.27	UV photo-oxidized, 0.2 μ filtered	656	145	1,979	311	19	13,449	623	300.1	20.4
D'	2.85	UV photo-oxidized, 0 ppm Cu, 200 ppm APDC	586	103	2,627	222	-6	12,017	473	2.33	2.4
D'	2.71	UV photo-oxidized, 10 ppm Cu, 200 ppm APDC	610	109	3,154	214	3,656	12,459	478	182.1	6.9
D'	2.57	UV photo-oxidized, 30 ppm Cu, 200 ppm APDC	574	94	2,862	102	982	11,982	383	9.39	5.3
D'	10.31	UV photo-oxidized, 0 ppm Fe, KOH, 0.5% PAC	626	116	347	61	14	13,495	701	55.69	31.5
D'	10.31	UV photo-oxidized, 10 ppm Fe, KOH, 0.5% PAC	624	120	508	80	67	13,099	651	23.07	8.2
D'	10.32	UV photo-oxidized, 30 ppm Fe, KOH, 0.5% PAC	613	111	526	32	11	11,295	584	22.29	10.0
D'	5.28	UV photo-oxidized, 1% iodated carbon	684	66	1,974	222	11	13,970	508	0.99	2.6
D'	4.22	UV photo-oxidized, 1% Keylex	405	128	885	307	17	13,793	431	17.78	28.3
D°	12.77	raw, unfiltered	674	126	4,089	1289	13	14,121	390	747	11.1
D°	12.77	raw 0.2 μ filtered	662	117	3,600	1125	11	14,183	397	1,008	4.3
D°	12.13	raw 0 ppm Cu, 200 ppm APDC	719	122	4,004	326	-17	14,811	641	250.4	2.3
D°	12.13	raw 10 ppm Cu, 200 ppm APDC	692	95	3,594	282	6982	14,470	623	316.9	5.9
D°	12.11	raw 30 ppm Cu, 200 ppm APDC	730	152	4,032	307	7613	14,697	647	20.74	2.9
D°	12.12	raw 0 ppm Fe, KOH, 0.5% PAC	694	124	3,712	246	19	14,899	665	24.09	15.4
D°	12.14	raw 10 ppm Fe, KOH, 0.5% PAC	698	96	3,590	234	18	15,157	723	11.83	12.0
D°	12.11	raw 30 ppm Fe, KOH, 0.5% PAC	680	108	3,574	230	12	14,916	693	9.96	10.5
D°	12.12	raw 1% iodated carbon	739	111	3,969	312	25	15,537	686	295.8	11.1
D°	12.09	raw 1% Keylex	725	149	4,077	294	16	15,783	689	491.4	12.8

Tables 7 and 8: Metals Removal Summary (BASF wastewater treatment, tasks 2-5)

analyzed by

Frontier Geosciences Inc. 414 Pontius North, Suite B Seattle, WA 98109

phone: 206-622-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com

			Metals Concentrations, µg/L (ppb)								
Waste	pH	Sample	V	Cr	Fe	Ni	Cu	As	Sb	Hg	Pb
		method blank-1	1,669	-202	-6,343	98	88	-373	-10.5	0.003	32.5
		method blank-2	1,670	-206	-6,393	94	49	-376	-10.7	0.003	30.1
		method blank-3	1,666	-212	-6,579	93	41	-381	-11.0	0.002	28.1
		method blank-4	1,666	-207	-6,529	95	48	-371	-10.7	0.002	29.6
		mean	1,668	-207	-6,461	95	56	-375	-10.7	0.003	30.0
		SD	2	4	111	2	21	5	0.2	0.001	1.8
		estimated MDL	6	13	333	6	64	14	0.6	0.002	5.5
B'	2.87	UV photo-oxidized, 30 ppm Cu, 200 ppm APDC rep 1	539	245	1,348	79	33	758	11.4	0.44	26.9
B'	2.87	UV photo-oxidized, 30 ppm Cu, 200 ppm APDC rep 2	563	256	1,472	75	66	777	12.2	0.43	29.8
B'	2.87	mean	551	251	1,410	77	49	768	11.8	0.43	28.3
B'	2.87	RPD (%)	4.4	4.3	8.8	5.5	66.4	2.5	6.1	1.4	10.3
		matrix spiking level	10,000	10,000	10,000	10,000	10,000	10,000	10,000	2.00	10,000
B'	2.87	UV'd, 30 ppm Cu, 200 ppm APDC + MS	10,472	10,104	11,438	9,641	9,735	10,309	9,361	2.50	10,492
B'	2.87	% recovery	99.2	98.5	100.3	95.6	96.9	95.4	93.5	103	104.6
B'	2.87	UV'd, 30 ppm Cu, 200 ppm APDC + MSD	10,114	9,648	10,540	9,298	9,326	10,350	9,413	2.44	10,548
B'	2.87	% recovery	95.6	94.0	91.3	92.2	92.8	95.8	94.0	100	105.2
B'	2.87	RPD (%)	3.5	4.6	8.2	3.6	4.3	0.4	0.6	0.5	0.5
B°	10.06	raw 0 ppm Fe, KOH, 0.5% PAC rep 1	564	261	1,651	270	23	870	14.0	18.49	36.0
B°	10.06	raw 0 ppm Fe, KOH, 0.5% PAC rep 2	541	257	1,492	256	16	820	14.1	18.86	26.7
B°	10.06	mean	553	259	1,571	263	20	845	14.1	18.68	31.3
B°	10.06	RPD (%)	4.2	1.7	10.2	5.6	35.5	5.8	0.8	2.0	29.5
		matrix spiking level	10,000	10,000	10,000	10,000	10,000	10,000	10,000	75.00	10,000
B°	10.06	raw 0 ppm Fe, KOH, 0.5% PAC + 10,000 MS	10,485	10,127	11,467	9,801	9,627	10,542	9,715	99.91	10,530
B°	10.06	% recovery	99.3	98.7	99.0	95.4	96.1	97.0	97.0	100.3	105.0
B°	10.06	raw 0 ppm Fe, KOH, 0.5% PAC + 10,000 MSD	10,352	9,886	11,221	9,738	9,453	10,269	9,740	93.42	10,415
B°	10.06	% recovery	98.0	96.3	96.5	94.8	94.3	94.2	97.3	99.6	103.8
B°	10.06	RPD (%)	1.3	2.4	2.2	0.6	1.8	2.6	0.3	0.5	1.1

Tables 7 and 8: Metals Removal Summary (BASF wastewater treatment, tasks 2-5)

analyzed by

Frontier Geosciences Inc. 414 Pontius North, Suite B Seattle, WA 98109

phone: 206-622-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com

		Metals Concentrations, µg/L (ppb)								
extract	Sample	V	Cr	Fe	Ni	Cu	As	Sb	Hg	Pb
TCLP	extraction blank #1	-119	10	221	1	53	-17	3	0.001	46
TCLP	extraction blank #2	-122	5	238	5	66	-8	3	0.000	21
TCLP	extraction blank #3	-125	20	147	2	66	-8	2	0.000	5
TCLP	mean	-122	12	202	3	62	-11	3	0.000	24
TCLP	SD	3	8	48	2	8	5	0	0.000	21
TCLP	estimated MDL	9	23	145	6	24	16	1	0.001	62
TCLP	APDC sludge	60	95	579	13	<24	9,051	82	1.28	<62
TCLP	spent Keylex-100 resin	49	29	182	61	<24	2,185	25	0.04	<62
TCLP	PAC/Fe(OH) ₃ sludge	< 9	44	7,044	339	<24	691	28	0.03	<62
TCLP	wet iodated carbon sludge rep 1	11	<23	252	33	<24	2,062	21	0.68	<62
TCLP	wet iodated carbon sludge rep 2	<9	<23	<145	42	<24	2,609	21	0.56	<62
TCLP	average wet iodated carbon sludge	<9	<23	156	38	<24	2,336	21	0.62	<62
TCLP	dry iodated carbon with 5,000 ppm Hg	<9	<23	<145	<6	<24	<16	<1	1,357	<62

		Metals Concentrations, µg/g (ppm, wet weight basis)								
extract	Sample	V	Cr	Fe	Ni	Cu	As	Sb	Hg	Pb
total metals	APDC sludge	24.0	10.3	282	44.7	2,860	334	28.9	78.89	3.7
total metals	spent Keylex-100 resin	12.3	1.8	43	3.85	1.8	45.8	0.45	12.59	0.8
total metals	PAC/Fe(OH) ₃ sludge	2.9	2.3	650	10.0	<1.6	14.8	0.41	31.54	2.4
total metals	wet iodated carbon sludge rep 1	4.8	0.7	47	2.15	<1.6	33.2	0.27	9.82	3.7
total metals	wet iodated carbon sludge rep 2	3.4	1.1	35	2.14	<1.6	20.3	0.20	7.91	<0.6
total metals	average wet iodated carbon sludge	4.1	0.9	41	2.15	<1.6	26.8	0.23	8.89	2.1
total metals	digestion blank #1	8.48	-0.41	14	0.44	2.77	-1.00	0.23	0.0001	0.5
total metals	digestion blank #2	8.48	0.01	23	0.47	2.47	-1.48	0.19	0.0001	0.2
total metals	digestion blank #3	8.48	-0.21	7	0.25	1.75	-2.25	0.21	0.0001	0.2
total metals	mean	8.48	-0.21	15	0.38	2.33	-1.58	0.21	0.0001	0.3
total metals	SD	0.00	0.21	8	0.12	0.52	0.63	0.02	0.0000	0.2
total metals	estimated MDL	0.00	0.63	23	0.36	1.6	1.9	0.06	0.0001	0.6

Table 9: BASF Groundwater Mercury Removal Experiments (Mercury Data Only)*analyzed using EPA Method-1631 by*

Frontier Geosciences Inc. 414 Pontius North, Suite B Seattle, WA 98109

phone: 206-622-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com

note: results with > 95% removal are highlighted in red, while those with final [Hg] < 1 µg/L are shaded with blue

Effect of Pre-Treatment Only							
label	waste	pre-treatment	additive 1	additive 2	pH	Hg, ng/L	% Hg removal
white	B	unfiltered	HCl		2.04	22,771	-0.8
white	B	filtered	HCl		2.04	1,980	91.2
white	B'	unfiltered	HCl	H ₂ O ₂ + UV	2.04	18,528	18.0
white	B'	filtered	HCl	H ₂ O ₂ + UV	2.04	161	99.3
white	B°	unfiltered	raw		10.26	22,500	
white	B°	filtered	raw		10.26	22,700	
white	D	unfiltered	HCl		1.93	787,188	10.3
white	D	filtered	HCl		1.93	7,415	99.2
white	D'	unfiltered	HCl	H ₂ O ₂ + UV	3.27	531,448	39.4
white	D'	filtered	HCl	H ₂ O ₂ + UV	3.27	300,120	65.8
white	D°	unfiltered	raw		12.77	747,000	
white	D°	filtered	raw		12.77	1,008,000	

Table 9: BASF Groundwater Mercury Removal Experiments (Mercury Data Only)*analyzed using EPA Method-1631 by*

Frontier Geosciences Inc. 414 Pontius North, Suite B Seattle, WA 98109

phone: 206-622-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com

note: results with > 95% removal are highlighted in red, while those with final [Hg] < 1 µg/L are shaded with blue

Co-Precipitation with Cu-APDC and 0.2 µ Filtration							
label	waste	pre-treatment	additive 1	additive 2	pH	Hg, ng/L	% Hg removal
blue	B	acidification	0 ppm Cu	200 ppm APDC	3.75	2,370	89.5
blue	B	acidification	10 ppm Cu	200 ppm APDC	3.54	1,271	94.4
blue	B	acidification	30 ppm Cu	200 ppm APDC	3.24	1,053	95.3
blue	B'	H2O2 + UV	0 ppm Cu	200 ppm APDC	2.91	485	97.9
blue	B'	H2O2 + UV	10 ppm Cu	200 ppm APDC	2.92	545	97.6
blue	B'	H2O2 + UV	30 ppm Cu	200 ppm APDC	2.87	433	98.1
blue	B°	raw	0 ppm Cu	200 ppm APDC	10.04	24,286	-7.5
blue	B°	raw	10 ppm Cu	200 ppm APDC	10.03	21,110	6.6
blue	B°	raw	30 ppm Cu	200 ppm APDC	10.01	5,478	75.8
blue	D	acidification	0 ppm Cu	200 ppm APDC	3.48	7,588	99.1
blue	D	acidification	10 ppm Cu	200 ppm APDC	3.29	2,899	99.7
blue	D	acidification	30 ppm Cu	200 ppm APDC	3.91	6,260	99.3
blue	D'	H2O2 + UV	0 ppm Cu	200 ppm APDC	2.85	2,331	99.7
blue	D'	H ₂ O ₂ + UV	10 ppm Cu	200 ppm APDC	2.71	182,110	79.2
blue	D'	H2O2 + UV	30 ppm Cu	200 ppm APDC	2.57	9,393	98.9
blue	D°	raw	0 ppm Cu	200 ppm APDC	12.13	250,357	71.5
blue	D°	raw	10 ppm Cu	200 ppm APDC	12.13	316,995	63.9
blue	D°	raw	30 ppm Cu	200 ppm APDC	12.11	20,736	97.6

Table 9: BASF Groundwater Mercury Removal Experiments (Mercury Data Only)*analyzed using EPA Method-1631 by*

Frontier Geosciences Inc. 414 Pontius North, Suite B Seattle, WA 98109

phone: 206-622-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com

note: results with > 95% removal are highlighted in red, while those with final [Hg] < 1 µg/L are shaded with blue

Co-Precipitation with Fe(OH) ₃ plus Powdered Activated Carbon and 0.2 µ Filtration							
labe	waste	pre-treatment	additive 1	additive 2	pH	Hg, ng/L	% Hg removal
orange	B	acidification	0 ppm Fe	0.5% PAC	11.29	14,228	37.0
orange	B	acidification	10 ppm Fe	0.5% PAC	11.15	14,757	34.7
orange	B	acidification	30 ppm Fe	0.5% PAC	10.93	15,256	32.5
orange	B'	H ₂ O ₂ + UV	0 ppm Fe	0.5% PAC	9.10	30	99.87
orange	B'	H ₂ O ₂ + UV	10 ppm Fe	0.5% PAC	8.81	33	99.85
orange	B'	H ₂ O ₂ + UV	30 ppm Fe	0.5% PAC	8.08	30	99.87
orange	B°	raw	0 ppm Fe	0.5% PAC	10.06	18,681	17.3
orange	B°	raw	10 ppm Fe	0.5% PAC	10.03	17,248	23.7
orange	B°	raw	30 ppm Fe	0.5% PAC	10.01	15,878	29.7
orange	D	acidification	0 ppm Fe	0.5% PAC	9.88	15,006	98.3
orange	D	acidification	10 ppm Fe	0.5% PAC	9.86	18,681	97.9
orange	D	acidification	30 ppm Fe	0.5% PAC	9.69	14,894	98.3
orange	D'	H ₂ O ₂ + UV	0 ppm Fe	0.5% PAC	10.31	55,695	93.7
orange	D'	H ₂ O ₂ + UV	10 ppm Fe	0.5% PAC	10.31	23,065	97.4
orange	D'	H ₂ O ₂ + UV	30 ppm Fe	0.5% PAC	10.32	22,291	97.5
orange	D°	raw	0 ppm Fe	0.5% PAC	12.12	24,099	97.3
orange	D°	raw	10 ppm Fe	0.5% PAC	12.14	11,830	98.7
orange	D°	raw	30 ppm Fe	0.5% PAC	12.11	9,962	98.9

Table 9: BASF Groundwater Mercury Removal Experiments (Mercury Data Only)*analyzed using EPA Method-1631 by*

Frontier Geosciences Inc. 414 Pontius North, Suite B Seattle, WA 98109

phone: 206-622-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com

note: results with > 95% removal are highlighted in red, while those with final [Hg] < 1 µg/L are shaded with blue

Adsorption onto Granulated Iodinated Carbon and 0.2 µ Filtration							
labe	waste	pre-treatment	additive 1	additive 2	pH	Hg, ng/L	% Hg removal
purple	B	acidification	1% IOC		4.30	1,395	93.8
purple	B'	H ₂ O ₂ + UV	1% IOC		4.19	159	99.3
purple	B°	raw	1% IOC		10.04	20,580	8.9
purple	D	acidification	1% IOC		4.00	2,331	99.7
purple	D'	H ₂ O ₂ + UV	1% IOC		5.28	997	99.89
purple	D°	raw	1% IOC		12.12	295,820	66.3
Adsorption onto Keylex-100 Sulfhydryl Resin and 0.2 µ Filtration							
labe	waste	pre-treatment	additive 1	additive 2	pH	Hg, ng/L	% Hg removal
red	B	acidification	1% Keylex		3.76	813	96.4
red	B'	H ₂ O ₂ + UV	1% Keylex		2.76	16	99.93
red	B°	raw	1% Keylex		10.04	7,626	66.3
red	D	acidification	1% Keylex		3.49	1,104	99.87
red	D'	H ₂ O ₂ + UV	1% Keylex		4.22	17,777	98.0
red	D°	raw	1% Keylex		12.09	491,375	44.0

Table 9: BASF Groundwater Mercury Removal Experiments (Mercury Data Only)*analyzed using EPA Method-1631 by*

Frontier Geosciences Inc. 414 Pontius North, Suite B Seattle, WA 98109

phone: 206-622-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com

note: results with > 95% removal are highlighted in red, while those with final [Hg] < 1 µg/L are shaded with blue

Analytical QC Data							
labe	waste	pre-treatment	additive 1	additive 2	pH	Hg, ng/L	% Hg removal
orange	B°	raw	0 ppm Fe	0.5% PAC	10.06	18,494	
orange	B°	raw	0 ppm Fe	0.5% PAC	10.06	18,868	
					mean	18,681	
					RPD (%)	2.0	
orange	B° + 75,000	raw	0 ppm Fe	0.5% PAC	10.06	99,913	
					% recovery	100.3	
orange	B° + 75,000	raw	0 ppm Fe	0.5% PAC	10.06	93,415	
					% recovery	99.6	
					mean	93,664	
					RPD (%)	0.5	
blue	B'	H ₂ O ₂ + UV	30 ppm Cu	200 ppm APDC	2.87	436	
blue	B'	H ₂ O ₂ + UV	30 ppm Cu	200 ppm APDC	2.87	430	
					mean	433	
					RPD (%)	1.4	

Table 9: BASF Groundwater Mercury Removal Experiments (Mercury Data Only)*analyzed using EPA Method-1631 by*

Frontier Geosciences Inc. 414 Pontius North, Suite B Seattle, WA 98109

phone: 206-622-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com

note: results with > 95% removal are highlighted in red, while those with final [Hg] < 1 µg/L are shaded with blue

Analytical QC Data (continued)							
labe	waste	pre-treatment	additive 1	additive 2	pH	Hg, ng/L	% Hg removal
blue	B' + 2,000	H ₂ O ₂ + UV	30 ppm Cu	200 ppm APDC	10.06	2,501	
					% recovery	103.4	
blue	B' + 2,000	H ₂ O ₂ + UV	30 ppm Cu	200 ppm APDC	10.06	2,439	
					% recovery	100.3	
					mean	2,470	
					RPD (%)	0.5	
yellow	B	raw	10 ppm Sn(II)	0-30 min purge	10.26	403	
yellow	B	raw	10 ppm Sn(II)	0-30 min purge	10.26	454	
					mean	429	
					RPD (%)	11.9	
yellow	B + 1,600	raw	10 ppm Sn(II)	0-30 min purge	10.26	1,986	
					% recovery	97.3	
yellow	D + 3,200	raw	10 ppm Sn(II)	0-30 min purge	12.77	4,629	
					% recovery	95.6	
yellow	D + 3,200	raw	10 ppm Sn(II)	0-30 min purge	12.77	4,833	
					% recovery	101.9	
					mean	4,731	
					RPD (%)	4.3	

Table 10: BASF Groundwater Mercury Removal Experiments (Mercury Data Only)*analyzed using EPA Method-1631 by*

Frontier Geosciences Inc. 414 Pontius North, Suite B Seattle, WA 98109

phone: 206-622-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com

note: results with > 95% removal are highlighted in red, while those with final [Hg] < 1 µg/L are shaded with blue

Reduction of Hg(II) to Hg ⁰ and Air Stripping							
lable	waste	pre-treatment	additive 1	additive 2	pH	Hg Purged, ng/L	% Hg removal
yellow	B	raw	none	0-30 min purge	10.26	-1	0.00
yellow	B	raw	none	30-60 min purge	10.26	92	0.41
yellow	B	raw	none	60-240 min purge	10.26	5	0.02
				sum 4 hour purge		96	0.42
yellow	B	raw	100 ppm Sn(II)	0-30 min purge	10.26	9,116	40.34
yellow	B	raw	100 ppm Sn(II)	30-60 min purge	10.26	430	1.90
yellow	B	raw	100 ppm Sn(II)	60-240 min purge	10.26	591	2.62
				sum 4 hour purge		10,137	44.85
yellow	B	raw	10 ppm Sn(II)	0-30 min purge	10.26	429	1.90
yellow	B	raw	10 ppm Sn(II)	30-60 min purge	10.26	14	0.06
yellow	B	raw	10 ppm Sn(II)	60-240 min purge	10.26	59	0.26
				sum 4 hour purge		502	2.22
yellow	B	acidification	100 ppm Sn(II)	0-30 min purge	5.5	2,266	10.03
yellow	B	acidification	100 ppm Sn(II)	30-60 min purge	5.5	760	3.36
yellow	B	acidification	100 ppm Sn(II)	60-240 min purge	5.5	1,909	8.45
				sum 4 hour purge		4,935	21.84

Table 10: BASF Groundwater Mercury Removal Experiments (Mercury Data Only)*analyzed using EPA Method-1631 by*

Frontier Geosciences Inc. 414 Pontius North, Suite B Seattle, WA 98109

phone: 206-622-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com

note: results with > 95% removal are highlighted in red, while those with final [Hg] < 1 µg/L are shaded with blue

Reduction of Hg(II) to Hg ⁰ and Air Stripping (continued)							
lable	waste	pre-treatment	additive 1	additive 2	pH	Hg Purged, ng/L	% Hg removal
yellow	D	raw	none	0-30 min purge	12.77	1	0.00
yellow	D	raw	none	30-60 min purge	12.77	3	0.00
yellow	D	raw	none	60-240 min purge	12.77	21	0.00
				sum 4 hour purge		25	0.00
yellow	D	raw	100 ppm Sn(II)	0-30 min purge	12.77	20,862	2.38
yellow	D	raw	100 ppm Sn(II)	30-60 min purge	12.77	10,346	1.18
yellow	D	raw	100 ppm Sn(II)	60-240 min purge	12.77	62,978	7.18
				sum 4 hour purge		94,186	10.73
yellow	D	raw	10 ppm Sn(II)	0-30 min purge	12.77	1,571	0.18
yellow	D	raw	10 ppm Sn(II)	30-60 min purge	12.77	20	0.00
yellow	D	raw	10 ppm Sn(II)	60-240 min purge	12.77	22	0.00
				sum 4 hour purge		1,613	0.18
yellow	D	acidification	100 ppm Sn(II)	0-30 min purge	5.5	734	0.08
yellow	D	acidification	100 ppm Sn(II)	30-60 min purge	5.5	219	0.02
yellow	D	acidification	100 ppm Sn(II)	60-240 min purge	5.5	776	0.09
				sum 4 hour purge		1,618	0.19

Table 11: Removal of Metals from Composite Well Water using Acidic KMnO₄ Oxidation and 2^o Treatment Options*experimental and analysis by*

Frontier Geosciences Inc. 414 Pontius North, Suite B Seattle, WA 98109

phone: 206-622-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com

	Metals Concentrations, µg/L (ppb)									
	Cr	Fe	Ni	Cu	Zn	As	Ag	Cd	Hg	Pb
D+B Initial Composite, pH 11.52 (a)	199	3,600	993	< 70	< 170	7,482	< 3	0.8	345.1	32
composite, KMnO ₄ + pH 1.44; settled 48 hours	109	< 1,900	266	< 70	< 170	6,859	< 3	< 0.4	4.41	< 4
composite, KMnO ₄ + pH 1.44; 0.2 µ filtered	110	< 1,900	243	< 70	< 170	6,809	< 3	< 0.4	1.80	< 4
treated filtrate + Co-APDC, 0.2 µ filtered	75	< 1,900	222	< 70	< 170	7,055	< 3	< 0.4	0.32	< 4
treated filtrate, 1% Keylex-100	92	< 1,900	236	< 70	< 170	7,022	< 3	0.9	0.08	< 4
treated filtrate + 0.2% PAC, 0.2 µ filtered	95	< 1,900	111	< 70	< 170	6,820	< 3	< 0.4	0.17	< 4
treated filtrate + 100 ppm Fe(OH) ₃ , 0.2 µ filtered	19	< 1,900	111	< 70	< 170	5,991	< 3	< 0.4	1.63	< 4
estimated MDL	11	1,900	4	70	170	2	3	0.4	0.001	4
 BASF Sludge µg/g (dry basis) (b)	 50.1	 1469	 24.9	 15.7	 39.5	 321	 0.35	 0.07	 161.6	 5.8
(a) calculated value as mean of previously measured initial concentrations										
(b) sludge from addition of KMnO ₄ and acidification only; dry fraction was 10.28% solids										

Table 12: Removal of Hg from Wells B+D Groundwater
a preliminary two-step removal concept designed for BASF by
Frontier Geosciences Incorporated

sample	pH	condition	[Hg], µg/L	% removal
B+D GW	11.52		1.31	
B+D GW	11.44	0.5% KMnO ₄ acidified 48 hrs	0.21	84.73
B+D GW	11.44	0.5% KMnO ₄ acidified 24 hrs	0.30	77.48
B+D GW acidified supernatant	11.15	200 rpm 24 hrs	0.12	90.81
B+D GW acidified supernatant	11.17	100 rpm 24 hrs	0.12	90.81
B+D GW acidified supernatant	11.17	0.22% powdered activated carbon	0.170	86.95
B+D GW acidified supernatant	11.87	100 rpm 24 hrs Fe(OH) ₃	0.153	88.53

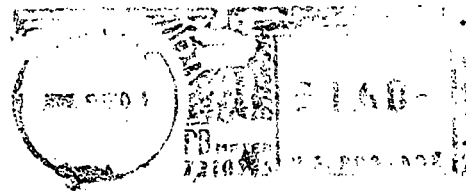
initial matrix constituents	mg/L	process step	sludge generated (g/L)
total dissolved solids	26,000	acidification plus KMnO ₄	8
total organic carbon	790	cobal FAPDC	0.5
free sulfide	22.2	powdered carbon	10
total cyanide	16.6	Fe(OH) ₃	3
iron	8.57	Keylex resin	0.1
manganese	0.02		

note: sludge generated @ 10% solids

BASF Corporation
1609 Biddle Avenue
Wyandotte, Michigan 48192

BASF

Amigo



Terese van Donzel
U.S. EPA
77 West Jackson Boulevard
SR-6J
Chicago, Illinois 60604